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MILITARY SPECIFICATION

PROPELLANT, MIXED OXIDES OF NITROGEN

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE.

1.1 Scope. This specification covers various mixtures of oxides of nitrogen propellants.

1.2 Classification. Mixed oxides of nitrogen propellants shall be of the following designated compositions as specified (6.2).

Compositions:

MON-10 - Nominal 90% N_2O_4 and 10% NO.

MON-25 - Nominal 75% N_2O_4 and 25% NO.

2. APPLICABLE DOCUMENTS.

2.1 Defense standardization documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of the specification to the extent specified herein.

SPECIFICATIONS

Military

MIL-P-25508	Propellant, Oxygen.
MIL-P-27401	Propellant Pressurizing Agent, Nitrogen.
MIL-P-27407	Propellant Pressurizing Agent, Helium
MIL-T-27730	Tape, Antiseize, Tetrafluoroethylene, with Dispenser

FSC 9135

STANDARDS

Military

MIL-STD-101	Color Code for Pipelines and for Compressed Gas Cylinders,
MIL-STD-129	Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

Department of Transportation (DOT)

49 CFR 170	Administrative procedures of the Department of Transportation.
49 CFR 171-190, 46 CFR 146, & 14 CFR 103.	Hazardous Materials Regulations of the Department of Transportation. (For use of alternate commercial publications see 6.8).

Department of Defense (DoD).

DSAM 4145.3; Also AFM 71-4, TM 38-250 NAVAIR 15-03-500 or MCO 4145.3.	Packaging and Handling of Dangerous Materials for Transportation by Military Aircraft.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.)

Manufacturing Chemists Association (MCA).

Manual L-1	Guide to Precautionary Labeling of Hazardous Chemicals (1970).

(Application for copies should be addressed to the Manufacturing Chemists Association, Inc., 1825 Connecticut Avenue, N.W., Washington, D.C. 20009.)

American Society for Testing and Materials (ASTM).

Recommended Practice

E 29-67 Indicating Which Places of Figures are to be Considered Significant in Specified Limiting Values.

Standards

D 2276-70 Particulate Contaminant in Aviation Turbine Fuels, Test for

E 203-64 Water Using Karl Fischer Reagent, Test for

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

2.2.1 Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal Agencies.

3. REQUIREMENTS

3.1 Chemical composition and physical properties. The chemical composition and physical properties of the propellants shall conform to those listed in table I when tested in accordance with the applicable test methods.

TABLE I. Chemical Composition and Physical Properties

Composition	Limits		Test Paragraphs
	MON-10	MON-25	
Nitrogen tetroxide assay (N_2O_4) (percent by wt	88.8 min	73.8 min	4.5.3
Nitric oxide (NO) assay (percent by wt)	11.0 max ~~10.0 min~~	26.0 max 25.0 min	4.5.2
Water equivalent (percent by wt)	0.17 max	0.17 max	4.5.4
Chloride (percent by wt) 1/	0.040 max	0.040 max	4.5.5
Particulate	10 mg/liter	10 mg/liter	4.5.6

1/ This test need not be performed on propellant manufactured by the ammonia-oxidation process.

3.2 Limiting values. The following applies to all specified limits of this specification. For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limitation value, in accordance with the rounding-off method of ASTM Recommended Practice, E 29-67, indicating which places of figures are to be considered significant in specified limiting values.

3.3 Filter. A filter with a 10 nominal and 40 absolute micron rating shall be installed between the manufacturer's plant and the container to be filled for delivery.

3.4 Sampler. The sampler shall be a double-ended container capable of accepting a 400-ml sample and constructed of 300 series stainless steel. The valves and any outlets or welds shall also be of 300 series stainless steel (6.3). Samples shall be taken in accordance with 4.4.2.16.

3.5 Qualitative. The propellant shall be a homogenous liquid when examined visually by transmitted light in accordance with 4.5.1.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements (6.2).

4.2 Classification of tests. The inspection and testing of the propellants shall be classified as quality conformance tests.

4.3 Test Conditions. The test conditions are described under the individual tests to which they apply.

4.4 Quality conformance tests. Quality conformance tests shall consist of:

(a) Individual tests 4.4.1

(b) Sampling tests 4.4.2

4.4.1 Individual tests. The propellant shall be subjected to the following test as described under 4.5:

Examination of product 4.5.1

4.4.2 Sampling tests. The propellant shall be selected in accordance with 4.4.2.1 and subjected (as appropriate to Compositions) to the following tests as described under 4.5:

(a) Nitric oxide assay 4.5.2

(b) Nitrogen tetroxide assay 4.5.3

(c) Water equivalent 4.5.4

(d) Chloride content 4.5.5

(e) Particulate 4.5.6

4.4.2.1 Sampling plan.

4.4.2.1.1 Lot. A lot shall consist of one of the following:

(a) The propellant produced in not more than 24 consecutive hours from a continuous process which is used to fill shipping containers directly from the process output. A continuous process shall be the production of product by continuous input of raw materials and output of finished product by one manufacturer in one plant with no change in manufacturing conditions or materials.

(b) The propellant from individual runs of a batch process which is used to fill shipping containers directly from the process output. A batch process shall be the production of product by runs from single additions of raw materials which are reacted and purified forming the product.

(c) The propellant from either or both the continuous and batch processes which is held in a single storage tank and subsequently withdrawn to fill shipping containers. The product shall be homogeneous at the time of withdrawal and shall not be added to while being withdrawn. After each addition to the storage tank, the contents shall constitute a separate lot.

4.4.2.1.2 Sample. A sample shall consist of not less than 400 milliliters (ml) of propellant. Two samples shall be taken for the tests specified in 4.4.2. One sample shall be used for tests (a) through (d) and the other for test (e). In the case of cylinder sampling, both samples shall be taken from the same cylinder. Unless otherwise specified, quality conformance tests shall be made of each sample of the propellant taken directly from the shipping containers (6.2). When required, two samples shall be forwarded to a laboratory designated by the procuring activity for subjection to the quality conformance specified herein.

4.4.2.1.3 Cylinders. The number of cylinders selected for sampling from each lot shall be in accordance with table II. The first and last containers to be filled within a given lot shall be sampled. Other samples may be selected at random. Each selected cylinder shall be thoroughly agitated immediately before sampling. The propellant from each container sampled shall constitute a separate sample.

TABLE II. Sampling for Test

Number of containers in lot	Number of containers to be sampled
1	1
2-8	2
9-27	3
28-64	4

4.4.2.1.4 Portable tanks, cargo tanks, and tank cars. A sample shall be taken from each portable tank, cargo tank, or tank car.

4.4.2.1.5 Other containers. Unless otherwise specified, other containers of 100 gallons or less water capacity (6.2) shall be sampled in accordance with 4.4.2.1.3. Containers greater than 100 gallons water capacity shall be sampled in accordance with 4.4.2.1.4.

4.4.2.1.6 Method of taking sample. Connect a 1/4-inch stainless steel tube approximately 1 1/2-feet in length to the container (cylinder or tank) dip-tube valve. Fit one end of the tube with a 1-inch to 1 1/4-inch pipe-thread bushing for attaching to the dip-tube valve and fit the other end with a 1/4-inch tee. Fit one leg of the tee with a 1/4-inch needle valve for purging and the other leg for connecting to the sampler inlet valve. Attach approximately 5 feet of polyethylene tubing to the purging valve to direct the propellant fumes away from the sampling area. Apply thread tape conforming to MIL-T-27730 to all thread connections. Wrap the tape under tension starting two threads back from the end and in the direction with the male pipe thread helix toward thread run-out. Wrap once plus an overlap of 1/2-inch at thread run-out end. Connect the evacuated sampler to the tee at an inclined 45-degree angle with the inlet valve down. Open the dip-tube valve and the purging valve. Purge until approximately 2,000 ml of propellant has been removed to clear the dip-tube. Then close the purging valve and open the sampler inlet valve. Open the outlet valve and continue sampling until liquid propellant appears. Close the sampler outlet, in-let, and dip-tube valves. Open the purging valve to clear the sample line and disconnect the sampler.

4.4.3 Rejection. When any sample of the propellant tested in accordance with 4.5 fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.5 Test Methods.

4.5.1 Examination of product. The sample (4.4.2.1.2) shall be examined with transmitted light to satisfy the qualitative requirements. Place 100 ml of sample in a clean, clear glass container (graduated cylinder or large test tube), cool in an ice-bath for 10 minutes, wipe dry, and examine visually by transmitted light from a light bulb or sunlight.

4.5.2 Nitric Oxide assay. The NO content of the sample shall be determined by the following method.

4.5.2.1 Tared sample. Evacuate and weigh to the nearest 0.001 gram (g) a sample bomb of approximately 60-ml capacity fitted with a needle valve. Support the sampler in a vertical position with the sample inlet valve down on a ring stand under a ventilated hood. Connect the bomb assembly to the sampler and provide for purging the connecting line. With all valves closed, open the purge and sampler valves. Purge the connecting lines until liquid sample emerges from the vent line, then close the purging valve. Open the bomb assembly valve and transfer 40 to 60 g of the sample. Close all valves and then open the purge valve to allow purging of remaining sample from the connecting lines. Disconnect the bomb assembly and flush the exposed opening of the valve with nitrogen until residual N_2O_4 disappears. Reweigh accurately the bomb assembly to the nearest 0.001 g obtaining the sample weight by difference. When sample weight is below 40 g, another sample shall be taken. Evacuation and reweighing of the sample bomb is unnecessary for subsequent fillings.

4.5.2.2 Procedure. Attach a rotameter with stainless steel tubing to the reducing valve of a cylinder of oxygen (O_2). Connect a length of 1/16-inch stainless steel tubing to the exit end of the rotameter. Open the cylinder valve and, purge residual air from the connecting lines and rotameter. Immediately attach the bomb assembly to the other end of the 1/16-inch tubing and using the reducing valve adjust the oxygen pressure to 30 pounds per square inch (psi). Close the cylinder valve. Place the bomb assembly in an ice bath and maintain the temperature at approximately 32°F (0°C). Permit O_2 to flow into the sample bomb by first opening the cylinder valve and then the bomb valve. Occasionally shake the bomb to hasten absorption and oxidation. Lack of O_2 flow through the rotameter indicates completion of reaction. Allow the bomb assembly to remain in the ice bath for 15 minutes with valves open after reaction has been completed. Disconnect the bomb assembly, remove from the bath, warm to ambient temperature and wipe dry. Place on a balance and record the weight to the nearest 0.001 g.

4.5.2.3 Calculation. The percent NO shall be calculated by the following formulas:

(a) $x = B-A$

(b) $y = C-B$

(c) NO, percent $\dfrac{[y-0.003857\,(z-\frac{x+y}{1.49})]\,187.5}{x} - 0.15$

Where:

x = sample weight in grams.

y = O_2 weight in grams.

z = volume of sample bomb in ml. Determined by difference in weight from evacuated and water filled.

A = weight of sample bomb in grams.

B = weight of sample bomb plus sample in grams.

C = weight of sample bomb plus sample and O_2.

4.5.2.4 Reagents and equipment. The following reagents and equipment or their equivalent shall apply as test conditions of 4.5.2:

(a) Reagents

(1) Nitrogen, MIL-P-27401, Type I

(2) Oxygen, MIL-P-25508, Type I

(b) Equipment

(1) Valve, needle, 1/8-inch NPT male inlet X 1/4-inch swagelock outlet, stainless steel.

(2) Bomb, sample, 2-inch diameter with 1/8-inch NPT female, 302 stainless steel, 24 gauge.

(3) Stand, ring.

(4) Tubing, polyethylene, length as required.

(5) Rotameter, glass-ball, with 0 to 1.0 liter per minute capability.

(6) Tubing, stainless steel, 1/16-inch diameter.

(7) Bath, ice.

(8) Balance, analytical, plus or minus 0.1 mg sentitivity, 0.1 mg accuracy.

4.5.3 Nitrogen tetroxide assay. The N_2O_4 content shall be performed on the oxidized 40 to 60 g N_2O_4 sample paragraph 4.5.2.1.

4.5.3.1 Preparation of sample. Cool the sample to approximately 32°F (0°C) with crushed ice. The cooled sample shall be introduced into a clean, tared (1 ml) glass ampoule directly from the sampler through a 1/16-inch stainless steel tube. The ampoule tip shall be inserted into an adequate size polyvinyl chloride tube, approximately 6 inches long. The ampoule is then suspended in a liquid difluorodichloromethane bath until frozen. Lift the frozen ampoule and wrap it with a piece of soft paper, removing the polyvinyl chloride tube. The ampoule shall be sealed in the hot flame of a H_2/O_2, or equivalent, torch by rotating at an angle. Wash, dry, and weigh the ampoule. Sample sizes shall average approximately 1.3 g where 0.5N alkali is used.

4.5.3.2 Procedure. The weighed, sealed ampoule shall be placed into a heavy-walled glass stoppered Pyrex bottle, or iodine flask, containing 100 ml of water plus 20 ml of 30% hydrogen peroxide. Chill contents in ice water. Lubricate the stopper with stopcock grease and insert it into the ground glass neck of the bottle and/or flask. The sealed ampoule shall be broken by vigorous shaking, keeping the stopper in place firmly. Shaking shall be continued until all brown fumes disappear. The bottle or flask is returned to the ice water to keep the contents cool, and shaken from time to time over a 15 minute period. The stopper shall be removed, rinsed off with distilled water into the contents of the bottle or flask and replaced with an air condenser. This assembly is removed from the ice water and placed on a steam bath or hot plate at moderate heat setting (approximately 120°C/250°F) for 45 minutes. The contents shall be allowed to cool to ambient temperature. Rinse the air condenser carefully and remove it. The broken glass of the ampoule shall be pulverized with the flat end of a heavy glass rod. The rod shall be rinsed with distilled water into the bottle or flask. Add 3 - 5 drops of methyl red indicator and titrate with 0.5N NaOH solution to the yellow end point. A reagent blank shall be run in the same manner as the sample.

4.5.3.3 Calculation. The weight percent N_2O_4 shall be calculated by one of the following formulas:

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N_2O_4, percent by weight = 1.534 (A-B)

where: $A = \frac{(a-b)N}{W} \times (3.00 + 0.016 \times \%\ NO)$

B = percent by wt. NO.

N = normality of NaOH,

W = grams of oxidized sample in ampoule.

a = ml NaOH for sample.

b = ml NaOH for blank.

4.5.3.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.3.

(a) Reagents

(1) Sodium hydroxide solution, 0.5N: Dissolve 20 g of ACS reagent grade, low carbonate, NaOH in distilled water in a 1000-ml volumetric flask. Allow to cool and dilute to the mark with distilled water. This solution shall be standardized with Standard 0.5N HCl to the methyl red end point. Transfer 30-50 ml standard HCl into a beaker from a 100-ml buret. Titrate the HCl with 0.5N NaOH using the same buret after cleaning. Store the 0.5N NaOH in a polyethylene container and insure the exclusion of carbon dioxide during storage.

(2) Distilled water.

(3) Methyl Red indicator solution: 0.1% by weight in distilled water.

(4) Hydrogen peroxide, ACS reagent grade, 30% by weight.

(5) HCl solution, 0.5N: The 0.5N HCl is standardized with primary standard grade diphenyl guanidine to the methy red end point.

(6) Diphenyl guanidine: Primary standard grade, G. F. Smith & Co., or equivalent.

(b) Equipment

(1) Buret, 100-ml, 0.2-ml graduation.

(2) Flask, iodine, 500-ml, heavy wall with 24/40 stopper.

(3) Ampoule, glass, 1-ml capacity.

(4) Beaker, 200 to 400 ml, as available.

(5) Flasks, volumetric, 1000-ml capacity.

(6) Ice bath.

(7) Low temperature bath, difluorodichloromethane or equivalent.

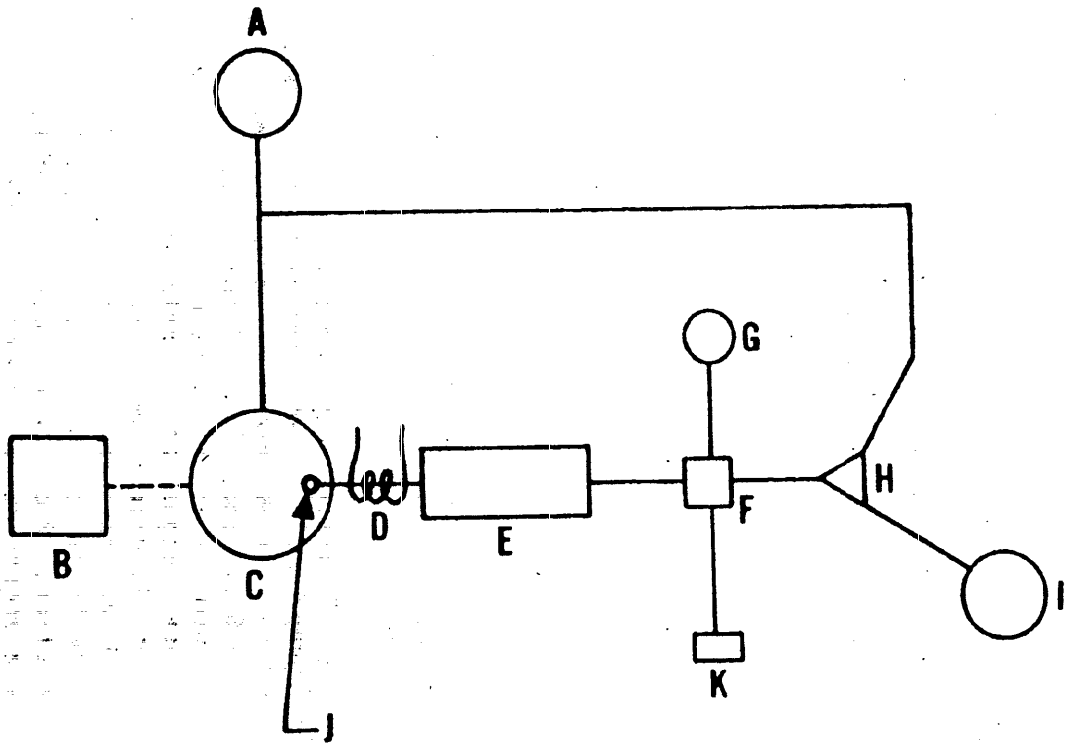
(8) Glass rod.

(9) Glass air condenser, 24/40 ground glass end with drip tip.

(10) Steam bath or hot plate.

4.5.4 Water equivalent. The water equivalent of the sample shall be determined by the following procedure:

4.5.4.1 Analytical apparatus. The analytical apparatus shall be prepared and assembled as shown schematically in figure 1. One side of a 2-µl stainless steel liquid inlet valve is connected to the three-way ball valve. The other side of the same 2-µl valve is connected to a 12-inch X 1/4-inch stainless steel tube packed with copper fillings and plugged with copper turnings or wool at each end. The copper-packed reactor is enclosed by a tube furnace capable of maintaining a constant temperature between 1112°F to 1202°F (600 to 650°C). The other end of the copper-packed reactor is connected to the sample inlet line of a gas chromatograph, containing a septum inlet with 1/16 or 1/8-inch OD stainless steel tubing. The gas chromatograph column shall be of sufficient length of tubing and contain a support material to adequately



LEGEND:

A– HELIUM SOURCE

B– RECORDER

C– GAS CHROMATOGRAPH

D– COOLING COIL

E– TUBE FURNACE

F– LIQUID INLET VALVE

G– N_2O_4 SAMPLE

H– THREE-WAY VALVE

I– HYDROGEN SOURCE

J– SEPTUM INLET

K–SCRUBBER (6.7)

Figure 1. Water Equivalent Test Apparatus Schematic

resolve nitrogen and water and methanol and water. The detector shall have a minimum sensitivity of 0.25 millivolt per microgram of water. The reference and sample helium flow rates in cc/min shall be matched within 5 percent. The column and detector temperatures and the helium flow rate shall be optimized to produce sharp peaks with adequate resolution. To regenerate the reactor, the three-way ball valve is connected to a hydrogen source. The chromatograph column is disconnected from the copper reactor. Regeneration is accomplished by alternating small amounts of hydrogen with the He flow into the apparatus until no more water can be detected at the outlet line of the reactor. The detector current is off during the regeneration because of the interrupted carrier flow. A chilled water line consisting of 1/8-inch copper tubing may be wrapped around the line between the furnace and the septum and also around that portion of the liquid injection valve through which the N_2O_4 sample will flow. Dead volume must be kept to a minimum throughout the system.

4.5.4.2 Procedure. The oxidized sample of 4.5.2 shall be connected to the 2-µl liquid valve of the gas chromatograph shown in figure 1. The apparatus specified in figure 1 shall be in an operational mode ready to analyze a 2-µl aliquot. Allow the oxidizer to flow through the valve to the N_2O_4 scrubber (6.7). When a steady stream of liquid flows, introduce the aliquot and stop the N_2O_4 flow. Repeat the analysis a second time. Determine the areas or the peak heights.

4.5.4.3 Calculation. The water equivalent shall be calculated as follows:

The peak heights or areas are averaged and the micrograms (µg) of water determined from a calibration graph previously prepared. The water equivalent content shall be calculated by the following formula:

$$\text{Water equivalent, percent by wt} = \frac{\mu g\ H_2O}{29}$$

The peak heights or areas of the duplicates must be reproducible within 2 percent. If they are not, premature sample vaporization or a contaminated valve may be responsible for variations.

4.5.4.4 Calibration. To correct for the original water present in the methanol, a calibration graph shall be prepared using aliquots of a standard methanol solution containing a known amount of water. Inject triplicate aliquots of 0.2 µl, 0.4 µl, 0.6 µl, and 0.8 µl of the standard solution with a 1.0-µl precision syringe into the septum inlet of the gas chromatograph. Average the areas or the peak heights of the triplicates and subtract the blank from each. This blank is determined first by filling and expelling 1.0 µl of the standard solution and then inserting the needle through the septum of the gas chromatograph. The heated septum inlet will vaporize a minute amount of the residual standard solution. Plot the corrected µg of H_2O versus the peak heights or area and draw a smooth curve through the origin.

4.5.4.5 Reagents and equipment. The following reagents and equipment or their equivalent shall apply as a test condition of 4.5.4:

a. Reagents

(1) Hydrogen, cylinder with regulator: Prepurified grade gas, 99.95%, 20 ppm O_2 maximum.

(2) Helium, gaseous, MIL-P-27407.

(3) Copper, 20-40 mesh: Pure, ACS reagent grade chips or filings, "Cuprin".

(4) Copper turnings: Pure, ACS reagent grade, degreased.

(5) Standard methanol/water solution: Absolute ACS reagent grade methanol, the water content of which shall be determined by ASTM Standard E 203-64. Add a sufficient, accurate amount of distilled water to make 500 ml of a 5 mg H_2O/ml solution. Verify the same ASTM method or equivalent. Keep tightly closed to prevent evaporation of methanol.

(6) Gas chromatographic column: Suggested columns and conditions are as follows:

(a) 6 to 10 ft of 5% polyethyleneglycol on polytetrafluoroethylene 40-60 mesh, at 158°F (70°C).

(b) 6 to 10 ft of "Porapack Q" at 212°F (100°C).

(c) Polyhalocarbon supports, polychlorotrifluorothylene with polyesters or polyglycols are satisfactory.

(d) Column shall be aluminum, copper, or stainless steel.

b. Equipment

(1) Tubing: Stainless steel, 1/8-inch, and 1/16-inch O.D. as required in figure 1.

(2) Cooling coils: 1/8-inch copper tubing O.D.

(3) Cooling bath: A source of 32 to 50°F (0-10°C) chilled fluid.

(4) Three-way valve: H_2-He supply, Hoke No. 7165G2S or 7165G4S.

(5) 2-µl liquid injection valve: Internal construction of stainless steel with polytetrafluoroethylene seals.

(6) N_2O_4 scrubber: N_2O_4 disposal shall be accomplished in accordance with applicable local anti-pollution laws.

(7) Gas chromatograph: A chromatograph with a thermal conductivity detector.

(8) Recorder: A potentiometric recorder with a 1-millivolt span and a 1-second FS response.

(9) Tube-furnace: 8 to 12 inches long with a 1/2 to 1-1/4-inch bore. Switch settings (voltage) shall be determined with a pyrometer so that it shall operate between 1112 to 1202°F (600 to 650°C). 500 to 1000 watts is adequate.

(10) Precision syringe: 1.0-µℓ capacity syringe graduated in 0.02-µℓ. Hamilton No. 7001 N with needle.

4.5.5 Chloride. The chloride content shall be determined by the following procedure.

4.5.5.1 Procedure. Weigh the sample bomb containing the oxidized sample prepared in 4.5.2.2 to the nearest 0.1g. Attach a polytetrafluoroethane tube to the bomb and to a gas washing bottle containing 50 ml of chloride-free distilled water. Slowly flow approximately 10g (7 ml) liquid into the gas wash bottle. Re-weigh the sample bomb. Quantitatively transfer the contents of the wash bottle to a 1000-ml volumetric flask. Add 2-5 drops of dinitrophenol and and neutralize with concentrated ammonia to the yellow endpoint. Dilute to the mark with chloride-free distilled water and mix. Pipet a 10.0 ml aliquot into a 25 ml volumetric flask. Add 10 ml of chloride-free distilled water and mix. Prepare a reagent blank with 20 ml of chloride-free distilled water in another 25 ml volumetric flask. Add exactly 2.0 ml of the ferric ammonium sulfate reagent to both and mix. Add exactly 1.0 ml of the saturated mercuric thiocyanate reagent and mix. Make up to the 25-ml marks with chloride-free distilled water and mix. Place both flasks in darkness for 15 to 30 minutes. Fill a 5-cm cell with the reagent blank and set the spectrophotometer at 0 absorbance (100%T) at 460 nanometer (nm). Measure the absorbance of the sample in a 5-cm cell at 460 nm. The mg Cl^- for the aliquot is determined from a calibration curve of absorbance vs mg Cl^-.

4.5.5.2 Calculation. The weight percent chloride shall be calculated as follows:

$$\text{wt. \%}Cl^- = \frac{(\text{mg } Cl^- \text{ from curve}) \times F \times 100}{\text{mg sample}}$$

where F = aliquot factor (i.e. 1000/10 etc).

4.5.5.3 Calibration. Pipet exactly 1.0, 2.0 and 4.0 ml of the dilute standard Cl^- solution into three 25-ml volumetric flasks. Add 15 ml of chloride-free distilled water. Transfer 20 ml chloride-free distilled water into a fourth 25-ml volumetric flask as a reagent blank. Add 2.0 ml of the ferric ammonium sulfate reagent to all flasks. Add exactly 1.0 ml of the saturated mercuric thiocyanate reagent and mix. Make up to the 25-ml marks with chloride-free distilled water and mix. Place both flasks in darkness for 15 to 30 minutes. Fill a 5-cm cell with the reagent blank and set the spectrophotometer at 0 absorbance (100%T) at 460 nm. Measure the absorbance of each calibration standard in a 5-cm cell at 460 nm. Plot the absorbances (y-axis) at 460 nm of the standards against mg Cl^- (x-axis) on linear graph paper, drawing a smooth curve through the origin.

4.5.5.4 Reagents and equipment. The following reagents and equipment shall apply as test conditions of 4.5.5.

a. Reagents

(1) Chloride-free distilled water: The water is considered chloride-free if a 100 ml sample, treated with silver nitrate, shows no turbidity during a 24-hour period, when stored in darkness.

(2) 2,4-dinitrophenol indicator, 0.1 wt. percent in alcohol and water.

(3) Ammonia, ACS reagent grade, 28 percent HN_3.

(4) Nitric acid: ACS reagent grade, 70 percent HNO_3.

(5) Ferric ammonium sulfate, 0.25M; Weight 60.275g of ferric ammonium sulfate dodecahydrate, $Fe\ NH_4\ (SO_4)_2 \cdot 12H_2O$, ACS reagent grade, and make up to 500 ml volume with 9M nitric acid. Filter or decant after 24 hours.

(6) Saturated mercuric thiocyanate: Dissolve 100 mg (0.10g) ACS reagent grade $Hg(SCN)_2$ in 100 ml 95 percent ethanol. Allow to stand, decant, and store in darkness.

(7) Stock Standard chloride solution: Dissolve 1.649g of dry Primary Standard grade sodium chloride in chloride-free distilled water, and dilute to the mark in a 500-ml volumetric flask. (1 ml = 2.0 mg Cl^-).

(8) Dilute standard Cl^- solution: Dilute 1.00 ml of reagent (7) to the mark in a 200-ml volumetric flask with chloride-free distilled water (1 ml = 0.010 mg Cl^-). Use a microburet to accurately measure 1.00 ml.

b. Equipment

(1) Chloride-free glassware: All glassware used must be washed with nitric acid and rinsed with chloride-free distilled water.

(2) Volumetric flasks, 25 ml, 100 ml, 200 ml, 500 ml, 1000 ml.

(3) Volumetric pipettes as required.

(4) Beakers, as required.

(5) Analytical balance, 0-200 gm, 0.1-mg sensitivity.

(6) 5.00-cm spectrophotometer cells.

(7) Spectrophotometer, capable of furnishing narrow band light between 400 to 500 nm, and capable of accepting 5.0-cm cells.

(8) Ringstand and clamps, as required.

(9) Microburet, 5.00-ml capacity.

(10) Gas wash bottles, 250-ml size,with or without fritted disk.

4.6 Preparation for delivery inspection. The packaging and marking of the propellant shall be inspected to determine compliance with the requirements of section 5 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The product furnished under this specification is a hazardous material as defined and regulated by the Department of Transportation (DOT) regulations. All packaging to be shipped commercially by any mode of transportation shall comply with the requirements of DOT regulations 49 CFR 171-190, or DOT special permit obtained in accordance with 49 CFR 170.13 by the shipper in conjunction with the Commander, Headquarters Military Traffic Management and Terminal Service, Attention: Safety Division (MTMTS/TA, Washington, D.C. 20315. All packaging to be shipped by military air shall comply with DSAM 4145.3 (AFM 71-4). The product shall be packaged in containers and unit quantities as specified by the procuring activity (6.2 and 6.3).

5.2 Preparation of containers. Prior to filling, the contractor shall determine the condition of all containers to ensure that they are free from contamination and suitable for shipment and storage. Contractor owned containers shall be cleaned and repaired by the contractor at his own expense. Leased or Government owned containers shall be cleaned and repaired in accordance with the schedule established in the contract or purchase order (6.2).

5.2.1 Cleaning and repair. Unless otherwise specified, all containers shall be visually inspected internally and externally for the presence of water, rust, scale and oil film, or other foreign matter, leakage, and physical damage (6.2). Any physical damage which would endanger safe transportation of the propellant shall be repaired prior to reuse. If evidence of internal contamination is found, the container shall be recleaned by a suitable method to remove the contamination.

5.2.2 Testing. Containers shall be hydrostatically or otherwise tested at a frequency specified by and in accordance with DOT requirements or as otherwise specified by the procuring activity.

5.2.3 Gaskets. Gaskets used to seal container openings shall be polytetrafluoroethylene or other material compatible with the propellant and approved for use by the procuring activity (6.2). The contractor shall assure that all gaskets are serviceable and furnish new gaskets when necessary so that a tight seal is assured.

5.3 Filling. The filling by density, weight, pressure, temperature, or any combination thereof shall be in accordance with DOT requirements or as otherwise specified by the procuring activity (6.2). Containers for which the prescribed tests have become due shall not be filled until retested.

5.4 Labeling and marking. Each container shall be labeled and placarded in accordance with MIL-STD-129 and established DOT requirements or DOT special permit. In addition, an identification tag, precautionary label, and container color code with name identification shall be used on each cylinder.

5.4.1 Identification tag. An identification tag impervious to climatic conditions shall be wired to the outlet port of each cylinder and shall contain the following information: Propellant name, specification, with revision letter, designated composition, FSN number, quantity, name of manufacturer, name of contractor (if different from manufacturer), lot number, and date of manufacture.

5.4.2 Precautionary label. A precautionary label prepared in accordance with MCA Manual L-1 shall be applied to each cylinder (6.6).

5.4.3 Container color code. Each cylinder shall be color coded in accordance with MIL-STD-101. The exact name identification to be marked on the outside of the cylinder shall be "Nitrogen Oxide Mixture". Any other name identification shall be obliterated by removing or overpainting.

6. NOTES

This section contains nonmandatory provisions only to assist both the contractor and buyer in the proper understanding and utilization of this specification.

6.1 Intended use. The propellants described by this specification are intended for use as oxidizers in rocket engines.

6.2 Ordering data. Purchasers should exercise any desired options offered herein, and procurement documents should specify the following:

6.2.1 Procurement requirements.

(a) Title, number, composition, and date of this specification (1.2).

(b) Method of shipment, type and capacity of containers (5.1 and 6.3).

(c) When other than quantity by weight in pounds (avoirdupois), state filling requirement (5.3).

(d) When inspection requirements are to be performed by other than the supplier (4.1).

(e) When waiver of quality conformance tests on each sample is granted(4.4.2.1.2).

(f) When unusual containers are to be used and sampled other than as specified (4.4.2.1.5).

(g) When cleaning and repair schedule is required for leased or Government owned containers (5.2).

(h) When cleaning and repair provisions are other than as specified (5.2.1).

(i) When hydrostatic test frequency is other than as specified (5.2.2).

(j) When approval of gasket material is required (5.2.3).

6.2.2 Contract data requirements. Data conforming to Data Item Description DI-T-138-1, Quality Conformance Test Reports (Fuels), is a requirement for delivery in connection with this specification. The data item will be specified for delivery on the DD Form 1423. In addition to the copies required by the procuring activity, one copy of each report should be addressed to AFRPL (DOZS), Edwards, CA 93523.

6.3 Containers. As of the date of this specification, the following listed containers are considered acceptable for military use and are approved for mixed oxides of nitrogen by DOT regulations as specified in 49 CFR 173.338, or DOT special permits as stated, or require action to obtain special permit as stated.

(a) Sample quantities should be shipped in cylinders conforming to MIL-C-83690, Type I, Style 1, High Pressure. Special permit required for these cylinders.

(b) Cylinders of specifications DOT 3A480, 3AA480, or individual cylinders of DOT 106A500.

(c) Tank cars of specifications DOT 106A500 or 106A500-X, or 105A500-W (with special requirements).

(d) Cargo tanks require action to obtain special permit.

6.4 Definitions.

(a) Particulate - The undissolved solids retained on a 10-micron filter membrane.

(b) Single-phased liquid - A single-phased liquid is devoid of any visible foreign liquid but may contain solid material as permitted within this specification.

6.5 Highway safety. To promote safety in the transportation of propellants in interstate commerce by motor vehicle, each product contractor or shipper should assure (and provide if necessary) that each driver possesses an MCA Chem-Card-Transportation Emergency Guide No. CC-1. A complete manual of cards or the individual cards are available from the Manufacturing Chemists' Association, 1825 Connecticut Avenue, N.W., Washington, D.C. 20009.

6.6 Precautionary labels. Precautionary labels are prepared in accordance with MCA Manual L-1. For those propellants which do not have specifically prescribed labels, the principles for the preparation of the labels are used. There does not have to be exact agreement between labels from different sources as long as the intent of the manual is complied with.

6.7 Pollution control. U.S. Public Laws dictate increased effort to improve air, land, and water pollution control of toxic propellant vapors, leaks, spills, and disposal during all phases of manufacture, transfer, storage, and transportation operations. The manufacturer/ supplier is enjoined to approach the appropriate pollution control district to mutually resolve all problem areas, and to develop adequate control and disposal methods for situations which are likely to develop in any of the phases.

6.8 Alternate publications. The following commercial publications may be used in lieu of or in conjunction with 49 CFR 171-190, 46 CFR 146, and 14 CFR 103 as appropriate as the content is identical or in the case of airlines is supplementary.

(a) Agent R. M. Graziano Tariff No. 23, Hazardous Materials Regulations of the Department of Transportation. Available from Association of American Railroads, Bureau of Explosives, 63 Vesey Street, New York, N.Y. 10007.

(b) Agent Fruend's Tariff, Hazardous Materials Regulations of the Department of Transportation. Available from American Trucking Association, Inc., 1616 P Street, N.W.,Washington, D.C. 20036.

(c) Tariff 6D, Transportation of Restricted Articles by Air. Available from Airline Tariff Publishers, Inc., 1825 K Street, N.W., Washington, D.C. 20006.

Custodians:

Army - MI

Navy - AS

Air Force - 12

Review Activities:

Air Force - 19,68, 70

Army - MI

Navy - AS

Preparing Activity:

Air Force - 12

Other Agency Interest:

NASA

Project No. 9135-0036

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

INSTRUCTIONS

This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity (as indicated on reverse hereof).

SPECIFICATION

MIL-P-27408A Propellant, Mixed Oxides of Nitrogen

ORGANIZATION (Of submitter)	CITY AND STATE

CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT
		$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A. GIVE PARAGRAPH NUMBER AND WORDING.

B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.

2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

☐ YES ☐ NO IF "YES", IN WHAT WAY?

4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)

SUBMITTED BY (Printed or typed name and activity)	DATE

DD FORM 1 APR 63 1426 REPLACES NAVSHIPS FORM 4863, WHICH IS OBSOLETE C-0879

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